# **BEST AVAILABLE COPY**

21 December 1989 (21.12.89) DK



## WORLD INTELLECTUAL PROPERTY ORGANIZATION International Bureau



#### INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification 5:

A23L 1/236, A23G 3/30, 3/00
A61K 9/68, 9/36

(11) International Publication Number: WO 91/09989

(43) International Publication Date: 11 July 1991 (11.07.9 H)

(21) International Application Number: PCT/DK90/00327
(22) International Filing Date: 12 December 1990 (12.12.90)

(22) International Filing Date: 12 December 1990 (12.12.90

(71) Applicant (for all designated States except US): DANSK TYGGEGUMMI FABRIK A/S [DK/DK]; P.O. Box 208, Dandyvej, DK-7100 Vejle (DK).

(72) Inventors; and (75) Inventors/Applicants (for US only): DYHR, Hans, Henrik [DK/DK]; Billensteinsvej 33, DK-7120 Vejle Øst (DK). SØRENSEN, Lene, Bothilde, Østergaard [DK/DK]; Barrit Langgade 96, DK-7150 Barrit (DK).

(74) Agent: CHAS. HUDE; H.C. Andersens Boulevard 33, DK-1553 Copenhagen V (DK).

(81) Designated States: AT, AT (European patent), AU, BB, BE (European patent), BF (OAPI patent), BG, BJ (OAPI patent), BR, CA, CF (OAPI patent), CG (OAPI patent), CH, CH (European patent), CM (OAPI patent), DE, DE (European patent), DK, DK (European patent), ES, ES (European patent), FI, FR (European patent), GA (OAPI patent), GB, GB (European patent), GR (European patent), HU, IT (European patent), JP, KP, KR, LK, LU, LU (European patent), MC, MG, ML (OAPI patent), MR (OAPI patent), MR, (OAPI patent), NO, RO, SD, SE, SE (European patent). SN (OAPI patent), SU, TD (OAPI patent), TG (OAPI patent), US, TD (OAP

Published
With international search report.

(54) Title: A METHOD FOR APPLYING A HARD COATING ON CORES OF ADIBLE OR CHEWABLE MATERIAL AND COATED MATERIAL OBTAINED BY THE METHOD

#### (57) Abstract

(30) Priority data:

6544/89

A method for applying a hard sorbitol coating to cores of edible or chewable material where the cores are in a moving bed. The cores are sealed by applying a sorbitol-containing suspension followed by smoothing out and drying in one or more cycles. Subsequently a forced hard coating with sorbitol suspension is carried out, followed by dry sorbitol powder in more cycles and finishing with a hard coating with sorbitol-containing suspension in one or more cycles. A dragee with a crunchingly crisp sorbitol coating is obtained.

### FOR THE PURPOSES OF INFORMATION ONLY

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AT	Austria	ES	Spain	MG	Madagascar
AU	Australia	FI	Finland	ML	Mali
BB	Barbados	FR	France	MN	Mongolia
BE	Belgium	GA	Gabon	MR	Mauritania .
BF	Burkina Faso	GB	United Kingdom	MW	Malawi
BG	Bulgaria	GN	Guinea	NL	Netherlands
BJ	Benin	GR	Greece	NO	Norway
BR	Brazil	HU	Hungary	PL	Poland
CA	Canada	IT	Italy	RO	Romania
CF	Central African Republic	J₽	japan	SD	Sudan
CG	Congo	KP	Democratic People's Republic	SE	Sweden
СН	Switzerland		of Korea	SN	Senegal
Ci	Côte d'Ivoire	KR	Republic of Korea	su	Soviet Union
CM	Cameroon	LI	Liechtenstein	TD	Chad
CS	Czechoslovakia	LK	Sri Lanka	TC	Togo
DE	Germany	LU	Luxembourg	US	United States of America
DK	Denmark	MC	Monaco		

PCT/DK90/00327

1

Title: A method for applying a hard coating on cores of edible or chewable material and coated material obtained by the method

#### 5 Technical Field

Control of the Control was under a control of the C

The present invention relates to a method for applying a hard sorbitol coating on cores of edible or chewable material, where the cores in a moving bed in a coating 10 apparatus are subjected to a number of coating cycles, each of the cycles comprising applying, distributing and smoothing out sorbitol-containing material and drying by means of drying air, whereupon the coated cores are optionally polished, as well as coated material obtained by 15 the method.

#### Background Art

It has been known for a long time to coat edible and 20 chewable materials with a sweet coating. Earlier such sweet coatings were usually based on sugar. In recent years a wish to make coated materials with sweet, sugarless coatings, for instance based on sorbitol as a sweetener has developed.

2.5

Within traditional sugar based coating techniques a distinction is made between different coating methods, for instance soft coating and hard coating. In principle, soft coating is based on the formation of a coating without substantial evaporation of water from the coating material added. This is done by adding both syrup or sugar solution and a dusting powder, whereby the material added forms a relatively soft coating on the cores without evaporation of water. In hard coating only a syrup or sugar solution is added, after the evaporation of water forming a coating harder than the one formed by soft coating. As a consequence of the water evaporation required, hard coating is

The Control of the Co

usually more time-consuming.

The crystallization of sorbitol is encumbered with problems due to the fact that there are more unstable crystal forms.

5 In crystallization of sorbitol from a solution an unstable amorphous form is firstly formed, said amorphous form subsequently being transformed via an unstable α-crystal form and an unstable β-crystal form to a stable gamma-crystal form. The final gamma-crystal structure is the one desired in a hard coating, as it ensures that the coating is provided with a pleasant crunchy consistency.

When moving from the traditional sugar-based coating technique to sugarless, for instance sorbitol-based, 15 coating, difficulties arise because sorbitol is difficult to crystallize, especially if the hard and crunchy gammacrystal structure is desired.

Several methods are already known for hard coating of 20 cores of edible or chewable material with a sorbitol coating. Thus, WO Publication No. 81/01100 discloses a method for hard coating with sorbitol, where cores placed on a moving bed are provided with a syrup having a concentration of dry matter of between 60 and 85% by weight 25 and with a D-sorbitol content based on dry matter of more than 80%, preferably more than 95%, and a temperature below 55°C. In this method, the sorbitol syrup must have a degree of saturation of between 0.65 and 1.25, preferably between 0.5 and 1.15 when the sorbitol syrup is contacted with 30 the cores to be coated. The coating is carried out in a conventional manner by means of succesive cycles, said cycles comprising a first step with the addition of the sorbitol syrup to the bed of cores and a second step wherein the addition is stopped while the rotation and 35 the prevailing temperature are maintained. The said second step is continued until the material applied during the first step is distributed over the cores and dried. The

gright respectively and the production of the second of the first second of the second of the second of the second

number of cycles determines the thickness of the coating obtained, there being narrow limits to the thickness of the layer applicable in each cycle if the coated product obtained is to have the desired attractive appearance. 5 Compared to the use of sugar for coating it is substantially more difficult to make a satisfactory sorbitol-coated product, because it is difficult to crystallize sorbitol to form the desired crystalline coating. To this should be added that chewing gum cores which are subjected to a 10 coating step with sorbitol easily stick to each other and form undesirable clumps. These difficulties are overcome by the method described in the above WO Publication, which provides a good hard, crunchy coating, which is desirable in an organoleptic quality product. The method is, however, 15 very time-consuming as the coating per se takes six hours to which should be added that the coated cores must be conditioned for a certain period of time before the final polishing.

20 US Patent No. 4 238 510 and BE Patent No. 884 317 (Cherukuri et al.) discloses a method for sorbitol coating wherein it is ensured that the sorbitol crystallizes. By this method, cycles are repeatedly carried out comprising (a) application of a first coating syrup containing sorbi-25 tol, an adhesion or binder component and a film-forming agent, (b) application of a dry dusting powder in the form of a mix comprising sorbitol in a powdered form, a moisture absorbing component, an anti-sticking component and a dispersing agent, (c) distributing and smoothing out 30 of the material applied and (d) drying of the layer of material formed on the cores. Following said treatment it is necessary to condition the coated cores for 1 to 3days to ensure the crystallization. After the conditioning another coating syrup is applied, said syrup containing 35 the same components as those contained in the powder mix but dispersed or dissolved in water. Said conditioning for 1 to 3 days takes place on trays and the transfer of

rijera (1980-1994) i sagriresti i 1900-ili (1900-eti) i i istoro aro o subsagritata prato, por o procesa i rozega, a escope e escepe 🦡

the coated cores from the coating apparatus to trays and back to the coating tray prior to the application of the second coating syrup is a time and labour-consuming process which results in a poor utilization of the coating apparatus. Furthermore, the drying time required in step (d) of the drying cycles is comparatively long which constitutes a further limitation to the capacity of the coating apparatus. In spite of the extensive time-consumption by the known method, the coating layer is not as crunchingly hard as the coating layer obtained according to WO 81/01100. This is however not aimed at, as a soft coating is desired.

US Patent No. 4 317 838 and GB Patent No. 2 115 672 (Che15 rukuri et al.) disclose a further development of the method disclosed in US Patent No. 4 238 510, wherein more detailed instructions as to the composition of the material applied and the use of drying air are given. Said method is more simple but large amounts of auxiliary agents are 
20 still used. Like the product obtained according to US 
Patent No. 4 238 510, the resulting product has a soft 
crystal structure in the coating.

More examples of soft and hard coatings with sorbitol are 25 known, for instance such as described in WO Publication No. 88/08671, JP 61249350 and JP 57107159 but these patent specifications do not state how a crispness as good or better than the crispness obtained by following the instruction of WO Publication No 81/01100 is obtained.

However, it has now been found that an even better sorbitol coating can be obtained, said coating being even more crunchingly crisp than the coating obtained according to

35 out substantially faster, whereby the coating costs are reduced drastically.

WO 81/01100 and at the same time the coating is carried

#### Disclosure of Invention

It is therefore the object of the present invention to provide a method for sorbitol coating by which a hard 5 coating shell with an improved crunchy crispness is obtained and wherein the entire coating process including the subsequent polishing, is carried out in one step in the coating apparatus without intermediate conditioning and wherein a high coating rate is obtained, calculated as 10 weight gain per surface area per time unit is obtained.

The above object is obtained by the inventive method for applying a hard sorbitol coating on cores of edible or chewable material where the cores in a moving bed in a coating apparatus are subjected to a number of coating cycles, each of the cycles comprising applying, distributing and smoothing out sorbitol-containing material and drying by means of drying air, whereupon the coated cores optionally are polished, which metod according to the invention is characterised in that in succession the coating cycles comprise:

- 1) a sealing wherein the cores are subjected to 1 to 6 cycles, each comprising the following steps (a), (c)25 and (d):
  - step (a), applying a sorbitol-containing suspension having a temperature of 15-25°C and containing at least 83% by weight of D-sorbitol based on dry matter.
  - step (c) distributing and smoothing out the applied material on the surface of the cores,
- step (d), drying the material deposited on the cores using drying air having a temperature of 20-40°C and a relative humidity of 8-30%,

WO 91/09989 PCT/DK90/00327

y na weng gan wangsala 1922 na 1822 natah 1822 na na na kawayen sa makala kanasa na kabah na makawa na kaban w

6

2) a forced hard coating, wherein the cores are subjected to one or more cycles, each comprising the steps (a), (b), (c) and (d), each step (a), (c) and (d) being as mentioned above and step (b) comprising the application of a dry sorbitol powder with a particle size of between 40 micrometer and 125 micrometer and a D-sorbitol content of at least 94% by weight based on dry matter, and

10

- 3) a hard coating, wherein the cores are subjected to one or more cycles, each comprising the above steps (a), (c) and (d).
- 15 By the method according to the invention a hard crunchy product is obtained, which in an Instron test has proved to be even more crunchingly crisp than a corresponding product prepared according to the method described in WO Publication No. 81/01100. The results of a comparative 20 Instron test also show that by the method according to the invention an improved adhesion to the chewing gum core is obtained. The entire method, including the subsequent polishing, may be carried out in a single sequence of process steps in the same coating apparatus during a 25 short time, for instance 100 to 160 minutes, preferably 115 to 145 minutes without intermediate conditioning. Coating may be carried out with a low content of auxiliary agents which is essential to the high organoleptic quality of the end product. A suspension containing about 10 % by 30 weight solid auxiliary agents, for instance talc powder and titan dioxide, or less, for instance about 5 to 2% by weight may be used, it has even been found that the method may be carried out without any content of for instance talc
- powder as anti-sticking component or dispersing agent 35 thereby providing particularly excellent organoleptic properties.

and the control of th

An essential feature of the method according to the invention is that said method is started with a sealing carried out in the same way as a conventional hard coating. The sealing is carried out with 1 to 6 cycles, preferably 3 cycles.

By the method according to the invention, step (d) may advantageously be carried out with drying air having a temperature of 26 to 30°C and a relative humidity of 9 to 15% in a quantity of 25 to 95 m<sup>3</sup>/hour per m<sup>3</sup> of the surface area of the cores thereby ensuring the short preparation time and the desired hard, crunchy coating.

The temperature of the suspension used in step (a) is 15 preferably 18 to 22 °C.

A slowly rotating coating apparatus with mixing blades is a suitable apparatus for carrying out the method according to the invention, said coating apparatus being provided 20 with perforations in the cylinder-shaped surface of the coating apparatus to provide drying air. Said form ensures the required supply of drying air. Said coating apparatus may for instance be a coating apparatus of the brand Dria 1200, available from Driam Metallprodukt GmbH & CO.KG. 25 Eriskirch, The Federal Republic of Germany. Such coating apparatus may be included in an assembly provided with a dehumidifier, for instance of the brand Munters, available from Karl Munters A/S, Farum Gydevej 89, 3520 Farum, Denmark.

30

The method according to the invention may advantageously be carried out in such a manner that during the sealing (1) the drying step (d) is carried out by blowing drying air in at the bottom of the drying pan and up through the bed of cores, and that during said forced hard coating (2), step (d) is carried out by blowing the drying air in from the top of the coating apparatus and down through the bed

of cores, and that during said hard coating (3) step (d) is carried out by blowing the drying air in at the bottom of the coating apparatus and up through the bed of cores. As a result, a good contact is ensured between the cores and the drying air. Furthermore, the carrying along with the air of the dusting powder, which has been added during the forced hard coating, is avoided.

The invention further relates to a dragee in the form of 10 an edible or chewable core with hard sorbitol coating prepared by the method according to the invention.

Examples of edible or chewable cores coatable by the method according to the invention include chewing gum, including 15 medical and semimedical chewing gum, pastilles, snack products, caramel, chocolate, dried fruits, dried vegetables, fruit bars, deshelled nuts, seeds, for instance almonds, sunflower seeds or pumpkin seeds, and medical products.

20

An essential feature of the present invention is that the sorbitol powder used as dusting powder is pure dry D-sorbitol powder without auxiliary agents.

- 25 Thus, the content of D-sorbitol in the dusting powder is at least 94% by weight based on dry matter, preferably at least 96% by weight, particularly preferred at least 99% by weight.
- 30 It is rather surprising that dusting powder with a low content of auxiliary agents and even entirely without auxiliary agents may be used, as a person skilled in the art on the basis of the teaching appearing from for instance Cherukuri's above specifications may get the im-
- 35 pression that considerable quantities of auxiliary agents, such as binding agent, are needed to ensure a sufficiently good adhesion to the chewing gum core.

WO 91/09989

9

医环门切除 医甲腺 的复数 化原锑酸 计记录器 医双头皮肤 医内侧皮膜 医多氏膜 计原列设计 医皮肤 医外胚

As the sorbitol solution to form part of the sorbitol-containing suspension applicable in step (a) by the method according to the invention, a sorbitol solution available in the trade may be used. Such sorbitol solution contains according to the specifications at least 68% by weight D-sorbitol, usually about 70% by weight. In the examples below said solution is designated as 70% by weight sorbitol solution.

10

The sorbitol suspension used preferably has a low content of auxiliary agents but may of course contain conventional auxiliary agents as required. Thus, the suspension may contain an anti-sticking component, for instance calcium 15 carbonate, talc powder or magnesium trisilicate, preferably talc powder. As mentioned above, the method according to the invention may be carried out entirely without the use of an anti-sticking component or with only a low content of anti-sticking component. Substances such as talc powder 20 or other anti-sticking components may also be used as dispersing agents but may also be left out or kept at a low level, if desired.

As mentioned, a high D-sorbitol content in the sorbitol25 containg suspension applied in step (a) is essential to
obtain the desired hard crunchingly crisp coating. Thus
the content thereof is at least 83% by weight, preferably
at least 92% by weight, particularly preferred at least
96% based on dry matter.

30

Contrary to this, in the case of soft coating, the sorbitol syrup used is a sorbitol solution with below 68% by weight D-sorbitol or with a higher content of additives of for instance hydrogenated disaccharides, such as maltitol, tri- and heptahydrogenated saccharides and the like. Such

35 tri- and heptahydrogenated saccharides and the like. Such additives, by soft coating used in quantites of from 10 to 35% by weight, prevent or render difficult the gamma-

WO 91/09989 PCT/DK90/00327

til der direktoria ette telebere i tittig et i til terebe bill bete betek er er

10

crystal formation as they directly inhibit the crystallization. By using said additives a coating layer is obtained which contains amorphous sorbitol. Such coating is classified as a soft coating which means to say that it is somewhat less hard than the hard coating prepared by the method according to the invention.

The method according to the invention may be carried out both with and without talc powder in the sorbitol-contain10 ing suspension applied during step (a). The addition of dispersing agents such as talc powder or calcium carbonate has no adverse effect on the crispness, but if too large quantities are used, such as exceeding 11 % by weight, a substantial deterioration of the mouthfeel and thereby 15 the entire perception takes place. By the method according to the invention the best organoleptic properties have been obtained with 2% by weight talc powder in the sorbitol-containing suspension. With quantities of talc powder of between 5 and 10% by weight the product still has a fair quality, but a somewhat poorer mouthfeel compared to the use of 2% by weight talc powder.

Colourants may be added in a conventional manner. In principle, any colourant which is approved for use in 25 foodstuffs may be used. Examples of such colourants include titan dioxide, chlorophyll, indigotine, betacarotene or mixtures thereof. The colourants are preferably added during the final hard coating.

30 Furthermore, flavouring agents in the form of both liquid and solid flavouring agents of both natural and synthetic origine may be added. Examples of flavouring agents may comprise peppermint oil, spearmint oil and mixtures thereof, menthol, citrus oils, such as orange oil, lime oil or lemon oil, fruit extracts or essences, such as extract or essence of pear, apple, cherry, raspberry, strawberry, plum, cherry plum, grape, pineapple, peach, fig or date.

WO 91/09989 PCT/DK90/00327

11

Corresponding synthetic flavouring agents with the same flavour may be used as a supplement or alternative to the above natural flavouring agents. The flavouring agents may be added to the coating shell at different times during 5 the method according to the invention, for instance between 1 and 3 times during the preparation. The first time may, for instance, be just after the sealing, the second time after the third cycle of the forced hard coating, in which case it may be in the form of a powdered flavoring agent 10 and the third time may be during the final smoothing hard coating. The quantities of flavouring agent used in the coating shell will be from 0.4 to 6% by weight, preferably 1.0% by weight calculated as a ratio of the total coating shell. Highly potent sweeteners, such as aspartame and 15 acesulfam-K may further be added. The quantity of said sweeteners may vary between 0.03 and 0.15% by weight, preferably 0.1% by weight calculated on the basis of the total coating shell.

- 20 After the coating per se a polishing is carried out, as mentioned, said polishing by the method according to the invention may advantageously be carried out in the same coating apparatus. At the polishing a polishing agent, for instance carnauba wax/talc powder, is added in a conventional manner, for instance in a ratio of 1 to 1, carnauba wax alone, vegetable oils or solutions of gum arabic or other types of vegetable rubber.
- The further scope of the applicability of the present invention will become apparent from the detailed description given hereinafter. However, it should be understood that the detailed description and specific examples, while indicating preferred embodiments of the invention, are given by way of illustration only, since various changes and modifications within the spirit and scope of the invention will become apparent to those skilled in the art from this detailed description.

CHENTOLA THE MERCHA HER LEV DO CARANTER PROPERTY AND DRIVED FOR A CONCURRANT CHANGE AND CARACTER

Fig. 1 illustrates the results of a comparison test between a chewing gum prepared by the method according to the invention (curves 1 and 2) and a chewing gum prepared by the method disclosed in WO 81/01100 (curves 3 and 4). The test was carried out on an Instron apparatus where the load necessary to maintain a constant rate at the cutting through of the chewing gum in KN in relation to time was measured. As it appears from the curves, a substantially better, crunchy crisp product is obtained by using the method according to the invention compared to prior art.

#### Best Mode for Carrying Out the Invention

15

The invention is illustrated further below by means of some examples.

#### Examples

20

The general method in the below examples is as follows:

Chewing gum cores prepared in the form of sheets are used as starting material, said sheets being rolled out to chewing gum cores each having a weight of about 1 g by means of a profiled roller. An assembly is used comprising a coating apparatus of the brand Dria 1200, available from Driam Metallproduckt GmbH & CO.KG, Eriskirch, the Federal Republic of Germany. Dria 1200 is adapted for coating of 50 kg of chewing gum cores corresponding to a surface area of 23 m<sup>2</sup> ± 5%. The assembly has computer control of dosing quantities, smoothing out times, drying times, air quantities, drying air temperatures and direction of air flow. The coating apparatus may be set at different rates of speed from 1 to 15 r.p.m.

In the coating process 50 kg of chewing gum cores are

(2) 对于一点、数据工具工程的设置数据等。但是是工具的设置的数据表面的表示。

filled in the coating apparatus which is set at a speed of 8 r.p.m.. During the rotation the chewing gum cores are separated from each other. Drying air is fed to the assembly and surplus talc powder added during the rolling of the chewing gum cores is removed from the chewing gum cores. The separation and air flow continue for about 5 minutes.

#### Sealing

10

The rotation speed of the coating apparatus is increased to 11 r.p.m. and the first dosing of the sorbitol solution is added. The quantity of sorbitol suspension added is 0.5 kg. Smoothing out is subsequently carried out, that is 15 rotation without air flow, where the suspension applied is smoothed out on all the chewing gum surfaces for about 0.6 minute. Drying then takes place for 5 minutes by blowing air towards the coating bed, that is from below, also designated counter flow. The above process is repeated 20 the neccesary number of times, for instance between one and six times. Said first cycles, where only sorbitol suspension is dosed, are designated sealing in the present specification.

#### 25 Forced hard coating

Subsequently a new type of coating is carried out, designated forced hard coating. Said hard coating comprises the same type of cycle as the sealing but with the addition of a further step after the application of the sorbitol solution, said step involving the introduction of a dusting powder in the form of a sorbitol powder. A cycle within said part of the coating process comprises firstly the application of about 1.2 kg sorbitol suspension and immediately following the distribution of the suspension in the course of 30-60 seconds, about 2.8 kg. sorbitol powder is dusted on by means of compressed air. During

IL MARKET A TOMOTION DESCRIPTION OF THE PROPERTY OF THE

the dusting of the dusting powder the rotation speed of the apparatus is reduced to 8 r.p.m.. When all the powder has been absorbed by the wet chewing gum cores after a smoothing out time of about 2 minutes, the rotation speed of the pan is increased to 11 r.p.m. and the chewing gum cores are dried by feeding drying air from the top of the chewing gum cores which is designated direct air flow or con-current flow. The drying time is about 5 minutes. This process is also repeated a suitable number of times, for instance about 5 times, until the desired weight increase has been obtained.

#### Hard coating

15 The coating process per se is completed by a conventional hard coating, said hard coating in principle being carried out in the same manner as the sealing. The rotation speed of the pan is 11 r.p.m. and the rotation speed is kept constant during the remaining portion of the process. The 20 sorbitol suspension is added in decreasing quantity, starting with 1.2 kg. per cycle. The smoothing out time is adapted thereto to ensure an even distribution. The air flow is reversed to counter flow and the drying time is about 5 minutes per cycle. Said process is repeated till 25 the required final weight has been obtained, usually 8 to 10 cycles.

#### Polishing

30 The coated cores are then polished with a polishing agent comprising carnauba wax/talc powder in a weight ratio of 1 to 1. 0.5 g polishing agent is used for 1 kg of coated cores. After the addition of the polishing agent, said polishing agent is distributed during rotation for 5 minutes. Drying air of a temperature of 10°C is then fed for 10 to 15 minutes. The coated chewing gum cores are now finished and ready for packing.

,是是全部的数据,这个证明性是特别,是否是<del>是数据的</del>的第三人称形式,更加是实现的数据的。但是是数据的特别是实现的。这是是实现的是是是是实现的。这

To provide the required low humidity of the drying air a dehumidifier of the brand Munters, available from Karl Munters A/S, Farum Gydevej 89, 3520 Farum, Denmark, is 5 used.

The 70% by weight sorbitol solution used in the examples contains according to an analysis 71.25% by weight D-sorbitol.

10

#### Example 1

The present example illustrates hard coating of chewing gum cores with sorbitol by the method according to the 15 invention.

50 kg of conventional chewing gum cores each having a weight of 1 g and corresponding to a total surface area of 23  $m^2 \pm 5$ % are coated using the following materials:

20

#### Suspension AI

70% by weight sorbitol solution 97.6% by weight Talc powder 2.0% by weight Titan dioxide 0.4% by weight

25

#### Suspension AII:

70% by weight sorbitol solution 94.6% by weight
Talc powder 2.0% by weight
30 Titan dioxide 0.4% by weight
Water 3.0% by weight

#### Powder

35 The sorbitol dusting powder has a purity of more than 99 % by weight D-sorbitol and a particle size of 40 micrometer to 125 micrometer. Said dusting powder is used without

WO 91/09989

16

auxiliary agents.

The coating is carried out as described below during a total of 17 cycles finishing with the polishing.

가는 보통하고 함께 있다면 하고 있다면 하고 있다면 하고 있다. 이 나는 그는 그는 그들은 그 그래는 그 전략이 그 전략이 되는 것을 보는 것이 되는 것이다.

5

#### Cycle 1-3, sealing

The coating comprises firstly a sealing of the cores by means of three coating cycles each comprising the application of 0.5 kg suspension AI having a temperature of 20°C ± 2°C, smoothing out for 30 seconds and drying for 5 minutes. The drying is carried out in counter flow. The drying air has a temperature of 28°C ± 2°C and a dew point of -7°C corresponding to 2.25 g water/kg air or a relative 15 humidity of 9.8%. The drying air is fed at a quantity of 25 m³/minute corresponding to 1500 m³/hour. Total time for the first three coating steps is 16 minutes.

#### Cycle 4-8, forced hard coating

20

A coating layer is subsequently applied by in the order for each cycle

- a) applying suspension AII having a temperature of 20°C  $\pm$  2°C,
  - b) applying powder.
  - c) distributing and smoothing out, and

30

- d) drying under the same conditions as during the sealing in cycle 1-3, apart from the drying air being fed in concurrent flow.
- 35 The quantities applied and the smoothing out and drying times are as follows.

	Cycle	Suspension	Powder	Smoothing	Drying
		AII (kg)	(kg)	out time	time
				(min)	(min)
	4	0.6	2.5	1.0	5.0
5	5	1.0	1.3	1.0	5.0
•	6	1.0	2.0	1.0	5.0
	7 .	1.2	2.7	2.0	5.0
	8	1.2	2.7	2.0	4.0

10 Total time consumption for cycles 4 to 8 is 37 minutes.

#### Cycle 9-17, hard coating

The coating is completed with a hard coating, each cycle 15 comprising

- a) applying suspension AII, with a temperature of 20°C  $\pm\ 2^\circ\text{C}$
- c) distributing and smoothing out, and 20
  - d) drying in counter flow.

In the individual cycles the following conditions are used:

25	Cycle	Suspension AII (kg)	Smoothing out time (min)	Drying time (min)
	9	1.2	0.6	5.0
	10-11	1.0	1.0	5.0
30	12-13	1.0	1.5	5.0
	14-16	0.7	1.3	5.0
	17	0.5	5.0	

The total time for cycles 9-17 is 57 minutes. The total 35 time from step 1 to and including step 17 is thus 110 minutes.

我们是要说。在这个人的生活是自己的心理等的,也是在"我们的生态"的,这种是不多少。 化二烷 医多元的 网络

Subsequently the coated cores are polished by means of a polishing agent comprising carnauba wax/talc powder in the weight ratio of 1 to 1. 0.5 g of the polishing agent is used for 1 kg of coated cores. After the addition of 5 the polishing agent, said polishing agent is distributed for 5 minutes during rotation of the coating apparatus. Drying air having a temperature of 10°C is fed for 10-15 minutes.

10 In said manner a smooth, coated chewing gum with a hard crunchy shell, polished and ready for packing is obtained. The finished product has a good harmony between sweetness and taste and at the same time a pleasant mouthfeel. Total coating time including the subsequent polishing in the 15 coating apparatus is 130 minutes.

#### Example 2

Substantially the same procedure as in Example 1 is repeat-20 ed using the same sorbitol dusting powder as in Example 1 and suspension AII for both the sealing, the forced hard coating and the conventional hard coating.

The coating is carried out as described below during a 25 total of 17 cycles finishing with polishing.

#### Cycle 1-3, sealing

In each of the three coating cycles 0.5 kg. suspension 30 AII is applied at a temperature of 20°C ± 2°C and smoothing out is carried out for 40 seconds and drying for 5 minutes. The drying is carried out in counter flow with a quantity of air, temperature and humidity as in Example 1. Total time for the first 3 coating cycles is 15 minutes.

#### Cycle 4-8, forced hard coating

Said coating cycles are carried out as in Example 1, the quantities applied and the smoothing out and drying times 5 being as follows:

	Cycle	Suspension	Powder	Smoothing	Drying
	•	AII (kg)	(kg)	out time	time
				(min)	(min)
10	4	1.0	2.6	1.5	5.0
	5	1.2	2.8	2.0	5.0
	6	1.2	2.8	2.0	5.0
	7	1.2	2.8	2.0	50
	8	1.2	2.8	2.0	5.0

15

Total time consumption for cycles 4-8 is 40 minutes.

#### Cycles 9-17, hard coating

20 The hard coating is carried out in the same manner as in Example 1, the following conditons being used in the individual cycles.

	Cycle	Suspension	Smoothing out	Drying time
25		AII (kg)	time (min)	(min)
	g ·	1.2	1.0	5.0
	•	1.0	1.5	5.0
	10-12	0.8	1.5	5.0
20	13-14 15-16	0.7	1.3	5.0
30	17	0.5	1.0	3.0
	Τ/	0.5	1.0	3.0

Total time consumption for cycles 9-17 is 60 minutes. The total time from step 1 to and including step 17 is thus 35 115 minutes.

The coated cores are then polished in the same manner as

WO 91/09989 PCT/DK90/00327

20

described in Example 1. Total coating time including the subsequent polishing in the coating apparatus is 130 minutes .

5 The finished product is an even and smooth dragee with a hard crunchy shell, polished and ready for packing. The finished product has a crunching, crisp initial chew with a good sweetness, mouthfeel and aroma release.

#### 10 Example 3

The coating described in Example 2 is repeated using the same sorbitol dusting powder but using suspension B instead of suspension AII.

15

#### Suspension B

70% by weight sorbitol solution	96.6% by weight
Titan dioxide	0.4% by weight
20 Water	3.0% by weight

The coating including the polishing may be carried out in 130 minutes.

25 The finished product has a good harmony between sweetness and taste and at the same time a pleasant mouthfeel. The coating is crisp and crunchy.

#### Example 4

30

The coating described in Example 2 is repeated using the same sorbitol dusting powder but using suspension C instead of suspension AII.

ligis els successivation de completa de la completa

#### Suspension C

70% by weight sorbitol solution	95.6% by weight
Talc powder	1.0% by weight
5 Titan dioxide	0.4% by weight
Water	3.0% by weight

The coating including the polishing may be carried out in 120 minutes.

10

The finished product has a good harmony between sweetness and taste and at the same time has a pleasant mouthfeel. The coating is crisp and crunchy.

#### 15 Example 5

The coating described in Example 2 is repeated using the same sorbitol dusting powder but using suspension D instead of suspension AII.

20

#### Suspension D

	70% by weight sorbitol solution	91:6%	Ъу	weight
	Talc powder	5.0%	Ъу	weight
2.5	Titan dioxide	0.4%	Ъу	weight
	Water	3.0%	bу	weight

The coating including the polishing may be carried out in 115 minutes.

30

The product is smooth, completely coated and with a hard crunchy shell. There is a good harmony between sweetness and taste but in an organoleptic evaluation of the product the talc powder content is perceived as a less pleasant,

35 floury mouthfeel.

#### Example 6

等的,这个性能的多点。\$P\$ (1962年) (\$P\$\$P\$从底,201

The coating described in Example 2 is repeated using the same sorbitol dusting powder but using suspension E instead of suspension AII.

5

#### Suspension E

	70% by weight sorbitol solution	85.8% by weight
	Talc powder	10.0% by weight
10	Titan dioxide	0.2% by weight
	Water	4.0% by weight

The coating including the polishing may be carried out in 130 minutes.

15.

The product is smooth, completely coated and with a hard crunchy shell. There is a good harmony between sweetness and taste but in an organoleptic evaluation of the product the talc powder content is perceived as a less pleasant,

20 floury mouthfeel to an even greater extent than the coated chewing gum prepared according to Example 5.

#### Example 7

25 The present Example illustrates hard coating of almonds with sorbitol by the method according to the invention.

50 kg of almonds are filled into the coating apparatus and the rotation is started at a speed of 10 r.p.m.. The com30 puter is set at the required values: dosing quantites, smoothing out times, drying times, air quantity, temperature of drying air and direction of air flow. The computer is also set at the number of cycles to be run during sealing, forced hard coating and the final hard coating, 35 respectively.

The suspension used has the following composition:

WO 91/09989

23

我是这种的对象,因此没有我们被他们的是是否的问题。她是是一定她的一点的。这是一个一个<mark>被要</mark>的是一致是他们的现在分词的现象。她是他们是是这一个是是这一个,他们

70% by weight sorbitol solution 95.5% by weight Titan dioxide 0.3% by weight Water 4.0% by weight 5 Indigotine 0.2% by weight

Sealing: 5 cycles.

Forced hard coating: 8 cycles are used which is necessary 10 to form an even surface capable of covering the rather uneven almonds.

Hard coating: 7 cycles providing a smooth hard coating.

15 Total coating time: 130 minutes.

Flavouring is added to the coating shell in the form of peppermint oil added to the last sealing layer.

20 The almonds are not polished as a surface is desired which is a little rough and feels slightly dusty.

The end product is of a dusty light blue colour and the almonds are evenly rounded and slightly dusty.

25

Suspension temperature:  $24^{\circ}C \pm 5^{\circ}C$ .

Temperature of drying air: 26°C ± 2°C

30 Relative humidity: 19% (at 26°C)

Quantity of air: 36 m<sup>3</sup>/minute.

#### Example 8

35

The present example illustrates hard coating of dried banana slices with sorbitol by the method according to the

BARRON - LANGE BARRON BERLIGHT GER DE BEGER DE EFFERENCE DANS BEKENDE DE LANGE DE LANGE DE LANGE DE LE BERLIGHE

invention.

40 kg of dried banana slices are filled into the coating apparatus. The density being low, there is only space for 5 40 kg of dried banana slices in the coating apparatus. At the same time there is a large surface area. The same method is used as the one used when coating almonds.

The suspension comprises:

10

70% by weight sorbitol solution 98.7% by weight Titane dioxide 0.1% by weight Water 1.0% by weight Caramel colourant (E150) 0.2% by weight

15

Suspension temperature: 20°C ± 2°C

Temperature of drying air: 28°C ± 2°C

20 Relative humidity: 10% (at 28°C)

Quantity of air: 24 m<sup>3</sup>/minute.

Chocolate flavouring is added to the shell as flavouring.

25

A low quantity of air is kept as the density of the product is low. Very low humidity is used to prevent the banana slices from turning soft. Polishing is carried out with carnauba wax/talc powder in a weight ratio of 1 to 1.

30

The product presents itself as crunchy, crisp pieces with a brown surface.

#### Example 9

35

The present example illustrates hard coating throat lozenges with sorbitol by the method according to the

BODE STORM OF A BODE WILLIAM OF A BODE A SECOND WAS A SECOND OF A SECOND OF A BODE A B

invention.

50 kg of throat lozenges (salt/liquorice) in the form of balls with a diameter of about 8 mm are filled into the 5 coating apparatus. Air having a temperature of about 15°C is fed in counter flow to immediately prevent the balls from sticking together. The same number of cycles as in Example 1 is used,

10 The suspension used comprises:

70% by weight sorbitol solution Titane dioxide Water 97.6% by weight
0.4% by weight
2.0% by weight

15

Suspension temperature: 20°C ±2°C

Temperature of drying air: 22°C ±2°C

20 Quantity of drying air: 36 m<sup>3</sup>/minute

Relative humidity: 9% (at 22°C)

Liquid liquorice flavour is added to the shell as flavour-25 ing.

The throat lozengers are polished after completed coating with carnauba wax/talc powder in the weight ratio of 1 to 1.

30

#### Comparison tests

A test was carried out at Bioteknisk Institut in Kolding, Denmark to test the sorbitol shell of a product according to the invention compared to a product prepared according to WO Publication No. 81/01100, that is ordinary coating without powder. The results appear from Fig. 1 where the

大型等的工程,全面的运动工程,但是是有数据的数据,然后的一点多数的关系,这是要数据的数据,这一个一种,更有效性,更有效性,更强强的基础的。

curves 1 and 2 are from tests with a product according to the invention with powder coating as described in Example 2, and curves 3 and 4 are from ordinary coating without powder as in WO Publication No. 81/01100.

5

In the test method used a dragee was placed over a slot whereupon it was cut through by means of a Verner-Bratxler knife at constant rate (50 mm/minute), said knife being a sheet of anodized aluminum with an angular recess where 10 all the edges are deburred. The force necessary to obtain the constant rate of speed was constantly registered.

As mentioned, four tests were carried out, curves 1 and 2 illustrating tests with products according to the invention 15 and curves 3 and 4 illustrate tests with products as described in WO Publication No. 81/01100. It can generally be stated that the steeper the curve until fracture, the stronger the perception of a "glass-like" hard surface, and that a more rounded or indented curve illustrates the 20 perception of a very "grainy" surface. The clearer fracture of the curve, the clearer the perception of a hard glass-like dragee shell, that is a feeling in the mouth of a very crunchy piece of chewing gum.

25 As appears from the Figure, curves 1 and 2 are almost identical with homogenous coating and clear crunch and probably a good adhesion to the core. Curve 3 shows a fairly crisp, vaguely hard outer shell with a fair adhesion to the core and curve 4 shows a crisp, not particularly 30 hard dragee shell and presumably poor adhesion to the core.

The invention being thus described, it will be obvious that the same may be varied in many ways. Such variations are not to be viewed as a departure from the spirit and scope 35 of the invention and all such modifications as would be obvious to one skilled in the art are intended to be included within the scope of the following claims.

数数比较微微的转形。 化环境中的 HTM 特殊的 1964年,1964

#### Claims

- 1. A method for applying a hard sorbitol coating to cores of edible or chewable material where the cores in a moving 5 bed in a coating apparatus are subjected to a number of coating cycles, each of the cycles comprising applying, distributing and smoothing out sorbitol-containing material and drying by means of drying air, whereupon the coated cores are optionally polished, c h a r a c t e r -i s e d 10 in that in succession the coating cycles comprise:
  - 1) a sealing wherein the cores are subjected to 1 to 6 cycles, each comprising the following steps (a), (c) and (d):

step (a), applying a sorbitol-containing suspension having a temperature of 15-25°C and containing at least 83% by weight of D-sorbitol based on dry matter,

step (c) distributing and smoothing out the applied material on the surface of the cores,

- step (d), drying the material deposited on the cores using drying air having a temperature of 20-40°C and a relative humidity of 8-30%,
- 2) a forced hard coating, wherein the cores are subjected to one or more cycles, each comprising the steps (a), (b), (c) and (d), each step (a), (c) and (d) being as mentioned above and step (b) comprising the application of a dry sorbitol powder with a particle size of between 40 micrometer and 125 micrometer and a D-sorbitol content of at least 94% by weight based on dry matter, and
  - 3) a hard coating, wherein the cores are subjected to

one or more cycles, each comprising the above steps (a), (c) and (d).

Moreover, and the control of the con

- 2. The method as claimed in claim 1, c h a r a c t e r-5 i s e d in that the coated cores are polished immediately after the coating cycles without intermediate conditioning of the coated cores.
- 3. The method as claimed in claim 2, c h a r a c t e r-10 i s e d in that the polishing is carried out in the same coating apparatus as used for the coating.
- The method as claimed in claim 1, c h a r a c t e r-i s e d in that said sealing is carried out by completing
   three cycles each comprising the steps (a), (c) and (d).
- 5. The method as claimed in claim 1, c h a r a c t e ris e d in that step (d) is carried out using drying air having a temperature of 26-30°C and a relative humidity 20 of 9-15% in a quantity of 25-95 m<sup>3</sup>/hour per m<sup>2</sup> of the surface area of the cores.
- 6. The method as claimed in claim 1, c h a r a c t e ri s e d in that the sorbitol-containing suspension used 25 in step (a) has a temperature of 18-22°C.
- 7. The method as claimed in claim 1, c h a r a c t e r-i s e d in that the method is carried out in a slowly rotating cylinder shaped coating apparatus with mixing 30 blades, said coating apparatus being provided with perforations in the cylinder shaped surface of the coating apparatus for drying air feed.
- 8. The method as claimed in claim 1, c h a r a c t e r35 i s e d in that during said sealing (1) the drying step
  (d) is carried out by blowing drying air in at the bottom
  of the coating apparatus and up through the bed of cores,

ka kanangan dina kapadasak mangalak na mining kamban mangalah na kakara di bina kakara di karana di kabanasa d

that during said forced hard coating (2), step (d) is carried out by blowing the drying air in from the top of the coating apparatus and down through the bed of cores, 5 and

that during said hard coating (3) step (d) is carried out by blowing the drying air in at the bottom of the coating apparatus and up through the bed of cores.

10

9. A dragee in the form of an edible or chewable core with a hard sorbitol coating, c h a r a c t e r i s e d in that it is prepared according to the method as claimed in any of the claims 1 to 8 above.

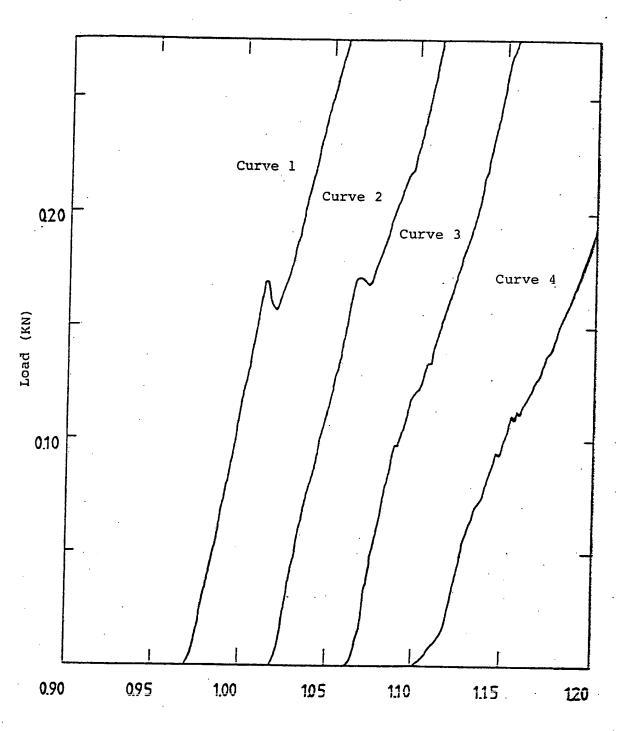
15

10. A dragee as claimed in claim 9, c h a r a c t e r - i s e d in that the core is of a material selected among the group consisting of chewing gum, pastilles, snack, caramel, chocolate, dried fruits, dried vegetables, fruit 20 bars, nuts, seeds and a medical products.

25

30

Fig. 1



Time (min.)

#### INTERNATIONAL SEARCH REPORT

International Application No PCT/DK 90/00327

I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all) <sup>6</sup>								
	According to International Patent Classification (IPC) or to both National Classification and IPC IPC5: A 23 L 1/236, A 23 G 3/30, 3/00, A 61 K 9/68, 9/36							
II. FIELDS SEARCHED								
	Minimum Documentation Searched <sup>7</sup>							
Classificat	Classification System Classification Symbols							
IPC5	A 23 L; A 23 G; A 61 K	·····						
	Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in Fields Searched 8							
SE,DK,F	FI,NO classes as above							
III. DOCU	MENTS CONSIDERED TO BE RELEVANT <sup>9</sup>	<del></del>						
Category *	Citation of Document, <sup>11</sup> with indication, where appropriate, of the relevant passages <sup>12</sup>	Relevant to Claim No.13						
Х	US, A, 4423086 (F. DEVOS ET AL.)	1-10						
	27 December 1983, see column 1,							
	line 25 - line 34; claim 1							
	<del></del>	. :						
x	WO AT SOMETH (UM UNITED BY IN COMPANY)	1-10						
^	WO, A1, 8808671 (WM. WRIGLEY JR. COMPANY) 17 November 1988, see claims 17-23,5	1-10						
[	Derwent's abstract, No. 82- 67 002/32, JP 57 107 159, publ. week 8232 (MORISHITA JINTAN KK)	1-10						
	Derwent's abstract, No. 86-334 847/51, JP 61 249 350, publ. week 8651 (KANEBO SHOKUHIN KK)	1-10						
1								
I								
"A" docu	categories of cited documents: 10  ment defining the general state of the art which is not idered to be of particular relevance  T later document published aft or priority date and not in co-cited to understand the principle.	er the international filing date nflict with the application but iple or theory underlying the						
"E" earling	er document but published on or after the international "X" document of particular releva	nce, the claimed invention						
"L" docui which citati	ment which may throw doubts on priority claim(s) or involve an inventive step	£ .						
"O" docu	cannot be constituted with a document is combined with a	lve an inventive step when the ne or more other such docu- ng obvious to a person skilled						
"P" docur	ment published prior to the international filing date but than the priority date claimed "&" document member of the sam	ne patent family						
. CERTIFI								
	Actual Completion of the International Search Date of Mailing of this International	Search Report						
	rch 1991 1991 -03- 2 6							
nernational	Searching Authority Signature of Authorized Officer							
	<u>SWEDISH PATENT OFFICE</u> Inga-Karin Peterssor							
m PCT/ISA/	/210 (second sheet) (January 1985)							

II. DOCL	OCUMENTS CONSIDERED TO BE RELEVANT (CONTINUED FROM THE SECOND SHEET)				
ategory •	Citation of Document, with indication, where appropriate, of the relevant passages	Relevant to Claim No			
	US, A, 4753790 (J. N. SILVA ET AL.) 28 June 1988, see claims 1,3,4,19	1-10			
	GB, A, 2115672 (NABRISCO BRANDS INC.) 14 September 1983, see page 1, line 3 - line 5; claims 1,11,13	1-10			
· .	US, A, 4238510 (S. R. CHERUKURI ET AL.) 9 December 1980, see claims 14-16,18,22	1-10			
	US, A, 4317838 (S. R. CHERUKURI ET AL.) 2 March 1982, see column 1, line 9 - line 14; claims 1-3	1-10			
.	•				
		· ·			
	*	•			
		-			
·					
	·				
	5 <b>9</b> 00				
		•			
	·	•			
·	j				
,					
	·				
-	·				

# BEST AVAILABLE COPY

# ANNEX TO THE INTERNATIONAL SEARCH REPORT ON INTERNATIONAL PATENT APPLICATION NO.PCT/DK 90/00327

This annex lists the patent family members relating to the patent documents cited in the above-mentioned international search report. The members are as contained in the Swedish Patent Office EDP file on 91-02-28 The Swedish Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

c	Patent document ited in search report			Publication date	
US-A-	4423086	83-12-27	AU-B- AU-D- CA-A- EP-A-B- WO-A- BE-A- CH-A- FR-A-B-	537832 6390580 1168082 0037407 81/01100 885778 645782 2467597	84-07-12 81-05-07 84-05-29 81-10-14 81-04-30 81-04-17 84-10-31 81-04-30
WO-A1-	8808671	88-11-17	AU-D- EP-A- JP-T- US-A-	1721288 0314739 2500483 4792453	88-12-06 89-05-10 90-02-22 88-12-20
US-A-	4753790	88-06-28	EP-A- JP-A-	0273856 63167744	88-07-06 88-07-11
GB-A-	2115672	83-09-14	NONE		
US-A-	4238510	80-12-09	NONE		
US-A-	4317838	82-03-02	AU-B- AU-D- BE-A- DE-A- FR-A-B- GB-A-B-	534011 6016680 884317 3025646 2486364 2079129	83-12-22 82-01-14 81-01-15 82-01-28 82-01-15 82-01-20

# This Page is Inserted by IFW Indexing and Scanning Operations and is not part of the Official Record.

### **BEST AVAILABLE IMAGES**

Defective images within this document are accurate representations of the original documents submitted by the applicant.

# IMAGES ARE BEST AVAILABLE COPY.

OTHER:

As rescanning these documents will not correct the image problems checked, please do not report these problems to the IFW Image Problem Mailbox.